TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

TestAmerica Laboratories, Inc.

# ANALYTICAL REPORT

## Perfluorocarbon (PFC) Analysis

**Lot #: D9J310192**


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# Case Narrative
**D9J310192**

TestAmerica Denver utilizes USEPA approved methods in all analytical work. The samples presented in this report were analyzed for the parameters listed on the methods summary page in accordance with the methods indicated. Dilution factors and footnotes are provided on each datasheet to assist in the interpretation of the results.

The results relate only to the samples in this report and meet all requirements of NELAC. All data have been reviewed for compliance with the laboratory QA/QC plan and have found to be compliant with laboratory protocols with any exceptions noted below.

Please note that Non-Detect (ND) results have been evaluated down to the Method Detection Limit (MDL) and should be considered ND at the MDL. Unless otherwise noted, results for solids have been dry weight corrected.

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**Sample Arrival and Receipt**
The following report contains the analytical results for one sample received at TestAmerica Denver on October 31, 2009, according to documented sample acceptance procedures. The sample was received in good condition at a temperature of 2.7°C. No anomalies were encountered during sample receipt.

**Standards**
Analytical standards were prepared using commercially available certified solutions containing all compounds of interest.

The mass labeled compounds 13C4 PFBA, 13C2 PFHxA, 18O2 PFHxS, 13C4 PFOA, 13C4 PFOS, 13C5 PFNA, 13C2 PFDA, 13C2 PFUnA, 13C2 PFDoA, and D3 MeFOSA were introduced at the extraction step and were used for internal standards for the quantitation of the target compounds.

**Sample Extraction and Analysis**
The samples presented in this report were extracted for the target analytes by TestAmerica Denver's Standard Operating Procedure (SOP) DV-OP-0019 and analyzed for the target analytes by TestAmerica Denver's SOP DV-LC-0012.

**Method QC Samples**
The Method Blank is processed reagent water spiked with internal standard and prepared with each batch of 20 samples of the same matrix. The method blanks were non-detect at the reporting limits for the target analytes.

Each batch is prepared with low and mid level Laboratory Control Samples (LCS). The LCS recoveries for both levels were within established control limits, with the exception of the items noted in section Analytical Comments.

**Analytical Comments**
The Standard Operating Procedure (SOP) was altered slightly in the sample preparation for FOSA. Sodium hydroxide was added to sample 115 1727 DENNIS MILL RD to obtain a pH of 14 instead of the SOP required <2. The basic pH is generating better internal standard recoveries for MeFOSA.

Due to an RPD failure in the mid-level LCS/LCSD and low percent recovery in the low-level LCS associated with batch 9309219, sample 115 1727 DENNIS MILL RD was re-extracted out of the laboratory prescribed hold time and reanalyzed in batch 9343491. Both batches have been included in this report. There is no prescribed regulatory holding time requirement for PFCs. The scientific literature indicates PFCs are highly persistent compounds in the environment. TestAmerica Denver has conducted stability studies indicating medium- and low-level standard solutions of PFOA are stable for at least three months in glass, polystyrene, and polypropylene plastics at 4±2 °C. The 7-day/40-day and 14-day/40-day holding times listed above are based on the general EPA convention for the holding time of extractable organic compounds in water and soil. Please note the sample results should be considered estimated.

The mid-level LCS/LCSD associated with QC batch 9309219 exhibited RPD data and/or a percent recovery outside the control limits for Perfluorooctanesulfonate (PFOS) and Perfluorodecane sulfonate (PFDS). The low-level LCS associated with QC batch 9309219 exhibited percent recoveries outside the QC control limits for Perfluorooctanesulfonate (PFOS), Perfluorohexanoic acid (PFHxA), and Perfluorononanoic acid (PFNA). Upon re-extraction and reanalysis in QC batch 9343491, percent recovery outliers were 100% in control. Both sets of data have been provided, as re-extraction was unavoidably performed outside the laboratory recommended sample holding time. Please note PFDS is not a compound of concern for this project.

Due to a limitation in the LIMS system, the low-level LCS associated with QC batch 9343491 reported the percent recovery for several Perfluorotridecanoic acid (PFTriA) as 0.0%. PFTriA was recovered within the control limits (50-150%) at 81%. As the compound was detected below the Method Detection Limit (MDL) of 0.01772 ug/L, the system reports the percent recovery as 0.0%.

The method required MS/MSD could not be performed for QC batches 9309211, 9309219, and 9343491, due to insufficient sample volume. Method precision and accuracy have been verified by the acceptable low-level LCS and mid-level LCS/LCSD analyses data.

The Standard Operating Procedure (SOP) was altered slightly for these samples in the sample prep and LC conditions. The alterations are listed below.

Solvents are now the same as they were in the original SOP and run per the following gradient: From 0 to 11 minutes, the flow rate is 0.4 mL/minute and the MeOH ramps up from 25% to 100%. From 11 to 11.01 minutes, the flow rate increases to 0.7 mL/minute and this flow is diverted from the MS. At 13 minutes the flow rate decreases back down to 0.4 mL/minute and 25% MeOH. The column then equilibrates to 14 minutes.

PFTriA and PFTeA now use 13C2 PFUnA as their internal standard instead of 13C2 PFDoA.

No other anomalies were observed.

# EXECUTIVE SUMMARY - Detection Highlights

**D9J310192**

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | ANALYTICAL METHOD |
|---|---|---|---|---|
| **115 1727 DENNIS MILL RD 10/29/09 14:31 001** | | | | |
| Perfluorooctanoic Acid | 0.012 J | 0.020 | ug/L | DEN -LC-0012 |
| Perfluorohexanoic acid (PFHxA) | 0.0034 J | 0.020 | ug/L | DEN -LC-0012 |
| Perfluorobutane sulfonate (PFB | 0.032 | 0.020 | ug/L | DEN -LC-0012 |
| Perfluorohexanoic acid (PFHxA) | 0.0046 J | 0.020 | ug/L | DEN -LC-0012 |
| Perfluorobutane sulfonate (PFB | 0.019 J | 0.020 | ug/L | DEN -LC-0012 |

# METHODS SUMMARY

**D9J310192**

| PARAMETER | ANALYTICAL METHOD | PREPARATION METHOD |
|---|---|---|
| LC/MS/MS PFCs | DEN -LC-0012 | SW846 FOSA spec |

**References:**


DEN Severn Trent Laboratores, Denver, Facility Standard Operating Procedure.

# METHOD / ANALYST SUMMARY

**D9J310192**

| ANALYTICAL METHOD | ANALYST | ANALYST ID |
|---|---|---|
| DEN -LC-0012 | Jacqueline Bonnett | 003601 |
| DEN -LC-0012 | Teresa L. Williams | 002510 |

**References:**

DEN Severn Trent Laboratores, Denver, Facility Standard Operating Procedure.

# SAMPLE SUMMARY

**D9J310192**

| WO # | SAMPLE# | CLIENT SAMPLE ID | SAMPLED DATE | SAMP TIME |
|---|---|---|---|---|
| LNM4X | 001 | 115 1727 DENNIS MILL RD | 10/29/09 | 14:31 |

**NOTE(S):**

- The analytical results of the samples listed above are presented on the following pages.
- All calculations are performed before rounding to avoid round-off errors in calculated results.
- Results noted as "ND" were not detected at or above the stated limit.
- This report must not be reproduced, except in full, without the written approval of the laboratory.
- Results for the following parameters are never reported on a dry weight basis: color, corrosivity, density, flashpoint, ignitability, layers, odor, paint filter test, pH, porosity pressure, reactivity, redox potential, specific gravity, spot tests, solids, solubility, temperature, viscosity, and weight.

Dalton Utilities

Client Sample ID: 115 1727 DENNIS MILL RD

HPLC

| | | | |
|---|---|---|---|
| **Lot-Sample #...:** D9J310192-001 | **Work Order #...:** LNM4X1AA | **Matrix.....:** WATER | |
| **Date Sampled...:** 10/29/09 14:31 | **Date Received..:** 10/31/09 | | |
| **Prep Date.....:** 11/05/09 | **Analysis Date..:** 11/12/09 | | |
| **Prep Batch #...:** 9309219 | **Analysis Time..:** 11:54 | | |
| **Dilution Factor:** 1 | | | |
| | **Method.....:** DEN -LC-0012 | | |

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | MDL |
|---|---|---|---|---|
| **Perfluorooctanoic Acid** | **0.012 J** | **0.020** | **ug/L** | **0.0098** |
| Perfluorooctanesulfonate | ND | 0.020 | ug/L | 0.013 |

| SURROGATE | PERCENT RECOVERY | RECOVERY LIMITS |
|---|---|---|
| 13C4 PFOA | 78 | (50 - 200) |
| 13C4 PFOS | 65 | (50 - 200) |

**NOTE(S):**

J Estimated result. Result is less than RL.

y

**Dalton Utilities**

**Client Sample ID: 115 1727 DENNIS MILL RD**

**HPLC**

| | | | |
|---|---|---|---|
| **Lot-Sample #...:** D9J310192-001 | **Work Order #...:** LNM4X2AA | **Matrix.....:** WATER | |
| **Date Sampled...:** 10/29/09 14:31 | **Date Received..:** 10/31/09 | | |
| **Prep Date.....:** 11/05/09 | **Analysis Date..:** 11/12/09 | | |
| **Prep Batch #...:** 9309219 | **Analysis Time..:** 11:54 | | |
| **Dilution Factor:** 1 | | | |
| | **Method.....:** DEN -LC-0012 | | |

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | MDL |
|---|---|---|---|---|
| Perfluoroheptanoic acid (PFHpA) | ND | 0.020 | ug/L | 0.013 |

| SURROGATE | PERCENT RECOVERY | RECOVERY LIMITS |
|---|---|---|
| 13C4 PFOA | 78 | (50 - 200) |

**Dalton Utilities**

**Client Sample ID: 115 1727 DENNIS MILL RD**

**HPLC**

**Lot-Sample #...:** D9J310192-001 **Work Order #...:** LNM4X3AA **Matrix.....:** WATER
**Date Sampled...:** 10/29/09 14:31 **Date Received..:** 10/31/09
**Prep Date.....:** 11/05/09 **Analysis Date..:** 11/16/09
**Prep Batch #...:** 9309219 **Analysis Time..:** 22:11
**Dilution Factor:** 1
**Method.....:** DEN -LC-0012

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | MDL |
|---|---|---|---|---|
| Perfluorobutanoic acid (PFBA) | ND | 0.020 | ug/L | 0.0098 |
| Perfluoropentanoic acid (PFPA) | ND | 0.030 | ug/L | 0.011 |
| **Perfluorohexanoic acid (PFHxA)** | **0.0046 J** | **0.020** | **ug/L** | **0.0029** |
| Perfluorononanoic acid (PFNA) | ND | 0.020 | ug/L | 0.017 |
| Perfluorodecanoic acid (PFDA) | ND | 0.020 | ug/L | 0.0078 |
| Perfluoroundecanoic acid (PFUnA) | ND | 0.020 | ug/L | 0.0069 |
| Perfluorododecanoic acid (PFDoA) | ND | 0.020 | ug/L | 0.015 |
| Perfluorotridecanoic acid (PFTriA) | ND | 0.020 | ug/L | 0.018 |
| Perfluorotetradecanoic acid (PFTeA) | ND | 0.020 | ug/L | 0.015 |
| **Perfluorobutane sulfonate (PFBS)** | **0.019 J** | **0.020** | **ug/L** | **0.0082** |
| Perfluorohexane sulfonate (PFHxS) | ND | 0.030 | ug/L | 0.0070 |

| SURROGATE | PERCENT RECOVERY | RECOVERY LIMITS |
|---|---|---|
| 13C4 PFOS | 61 | (50 - 200) |
| 13C4 PFBA | 85 | (50 - 200) |
| 13C2 PFHxA | 67 | (50 - 200) |
| 18O2 PFHxS | 110 | (50 - 200) |
| 13C5 PFNA | 103 | (50 - 200) |
| 13C2 PFDA | 80 | (50 - 200) |
| 13C2 PFUnA | 50 | (50 - 200) |
| 13C2 PFDoA | 54 | (50 - 200) |

**NOTE(S):**

J Estimated result. Result is less than RL.

**Dalton Utilities**

**Client Sample ID: 115 1727 DENNIS MILL RD**

**HPLC**

**Lot-Sample #...:** D9J310192-001 **Work Order #...:** LNM4X1AC **Matrix.....:** WATER
**Date Sampled...:** 10/29/09 14:31 **Date Received..:** 10/31/09
**Prep Date.....:** 11/05/09 **Analysis Date..:** 11/07/09
**Prep Batch #...:** 9309211 **Analysis Time..:** 00:02
**Dilution Factor:** 1

**Method.....:** DEN -LC-0012

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | MDL |
|---|---|---|---|---|
| Perfluorooctane sulfonamide (FOSA) | ND | 0.050 | ug/L | 0.0057 |

| SURROGATE | PERCENT RECOVERY | RECOVERY LIMITS |
|---|---|---|
| MeFOSA | 62 | (50 - 200) |

**Dalton Utilities**

**Client Sample ID: 115 1727 DENNIS MILL RD**

**HPLC**

**Lot-Sample #...:** D9J310192-001 **Work Order #...:** LNM4X1AD **Matrix.....:** WATER
**Date Sampled...:** 10/29/09 14:31 **Date Received..:** 10/31/09
**Prep Date.....:** 12/09/09 **Analysis Date..:** 12/29/09
**Prep Batch #...:** 9343491 **Analysis Time..:** 13:32
**Dilution Factor:** 1
**Method.....:** DEN -LC-0012

| PARAMETER | RESULT | REPORTING LIMIT | UNITS | MDL |
|---|---|---|---|---|
| Perfluorooctanoic Acid | ND | 0.020 | ug/L | 0.0098 |
| Perfluorooctanesulfonate | ND | 0.020 | ug/L | 0.013 |
| Perfluorobutanoic acid (PFBA) | ND | 0.020 | ug/L | 0.0098 |
| Perfluoropentanoic acid (PFPA) | ND | 0.030 | ug/L | 0.011 |
| **Perfluorohexanoic acid (PFHxA)** | **0.0034 J** | **0.020** | **ug/L** | **0.0029** |
| Perfluoroheptanoic acid (PFHpA) | ND | 0.020 | ug/L | 0.013 |
| Perfluorononanoic acid (PFNA) | ND | 0.020 | ug/L | 0.017 |
| Perfluorodecanoic acid (PFDA) | ND | 0.020 | ug/L | 0.0078 |
| Perfluoroundecanoic acid (PFUnA) | ND | 0.020 | ug/L | 0.0069 |
| Perfluorododecanoic acid (PFDoA) | ND | 0.020 | ug/L | 0.015 |
| Perfluorotridecanoic acid (PFTriA) | ND | 0.020 | ug/L | 0.018 |
| Perfluorotetradecanoic acid (PFTeA) | ND | 0.020 | ug/L | 0.015 |
| **Perfluorobutane sulfonate (PFBS)** | **0.032** | **0.020** | **ug/L** | **0.0082** |
| Perfluorohexane sulfonate (PFHxS) | ND | 0.030 | ug/L | 0.0070 |

| SURROGATE | PERCENT RECOVERY | RECOVERY LIMITS |
|---|---|---|
| 13C4 PFOA | 122 | (60 - 155) |
| 13C4 PFOS | 67 | (45 - 130) |
| 13C4 PFBA | 105 | (36 - 130) |
| 13C2 PFHxA | 108 | (55 - 135) |
| 18O2 PFHxS | 83 | (61 - 130) |
| 13C5 PFNA | 83 | (54 - 132) |
| 13C2 PFDA | 72 | (53 - 130) |
| 13C2 PFUnA | 72 | (37 - 130) |
| 13C2 PFDoA | 62 | (26 - 130) |

**NOTE(S):**

J Estimated result. Result is less than RL.